Atty. Docket No. SP-0649.1 (EVE01 P-565)

#24
6.2.04

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Art Unit	:	1745
Examiner	:	C. Chaney
Applicant	:	John C. Nardi
Appln. No.	:	09/213,544
Filing Date	:	December 17, 1998
For	:	Alkaline Cell Having A Cathode Incorporating Enhanced Graphite

Assistant Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

DECLARATION UNDER 37 C.F.R. §1.132

I, the undersigned, hereby declare:

1. I am the inventor of the above-identified patent application.

2. I am employed by the assignee in the above-identified application, Eveready Battery Company, Inc.

3. I executed a declaration under 37 C.F.R. §1.132 in the above-identified application on October 16, 2000.

4. I have thoroughly reviewed U.S. Patent No. 5,482,798 ("the Mototani et al. patent").

5. I believe that at least one of the commercially available expanded graphites (Samples B-D) in my October 16, 2000, declaration was made using a process that falls within the general description of how expanded graphite is made according to the Mototani et al. patent, to the extent that the process is disclosed therein. I believe this more clearly demonstrates that expanded graphite made according to the teachings set forth in Mototani et al. does not necessarily have a kerosene absorption value in the range of 2.2 to 3.5 ml/g.

6. In my October 16, 2000, declaration, I was not able to disclose details of the manufacturing processes for any of Samples B-D, because it was my understanding that such information had been provided to me by the manufacturers under confidentiality agreements between my employer and each of those manufacturers. Since I made my October 16, 2000,

declaration, a co-worker discovered in his files a publicly available brochure from Chuetsu Graphite works Co., Ltd., the supplier of the expanded graphite material identified as "Sample D" in that declaration. This brochure, attached as an Exhibit hereto, provides a general description of the process Chuetsu used to make expanded graphites (Special Treated Graphite on the eighth page of the Exhibit).

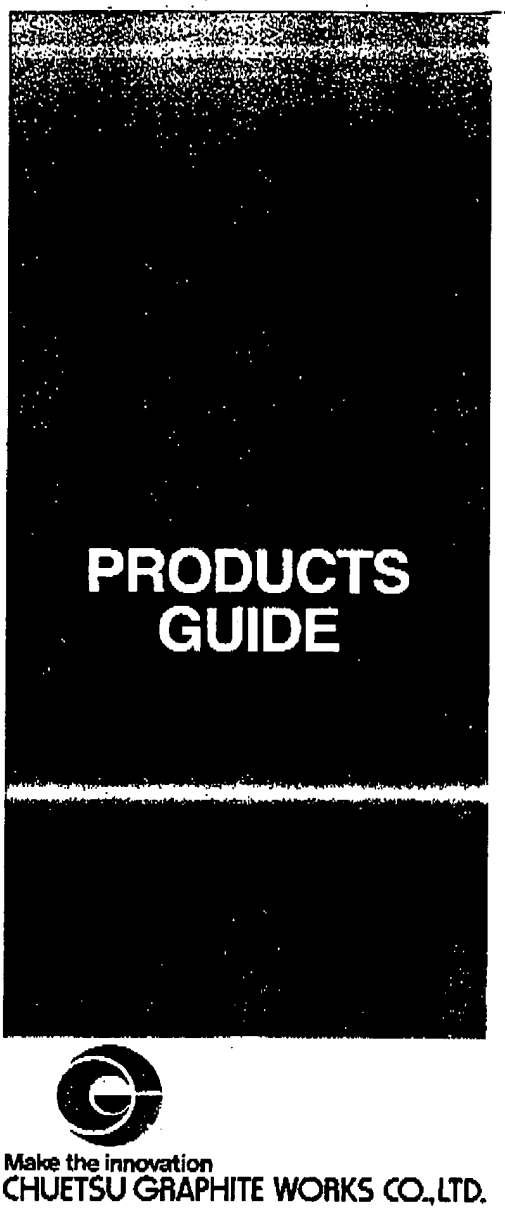
7. Chuetsu's brochure discloses, in part, that "flake graphite is treated with concentrated sulfuric acid ... heated at high temperature of 950° to 1100°C" to expand the graphite, which is then pulverized to make special treated graphite. Chuetsu's process employs common general process steps corresponding to those taught in Example 1 of the Mototani et al. patent, wherein expanded graphite is prepared by introducing sulfuric acid into between interlayers of the artificial graphite and heating rapidly at a temperature of 800° to 1000°C to expand greatly spaces between the interlayers of the graphite, and the resultant expanded graphite is crushed (col. 3, lines 40-51).

8. Sample D in the Table in my October 16, 2000, declaration exhibited a kerosene absorption value of 1.98 ml/g. The Sample D material was identified to me by Chuetsu as BSP-grade material. It is my understanding that the Sample D material was made according to the same general method as the special treated graphites (also BSP grades) listed on the sixth page of the Exhibit, and therefore, expanded graphite made according to the general teachings of the Mototani et al. patent would not necessarily have a kerosene absorption value of 2.2 to 3.5 ml/g.

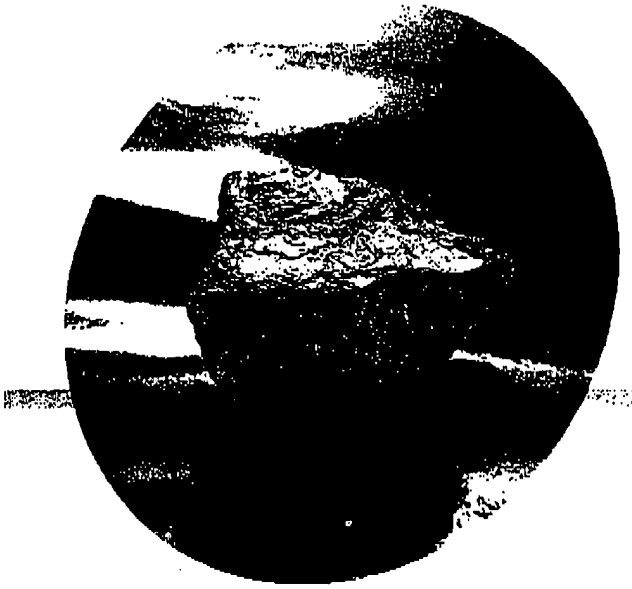
The undersigned hereby declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Sections 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

By: John C. Nardi
John C. Nardi

2-5-2003
Date



EXHIBIT



次代のために、無限
黒鉛パワーを世界

黒鉛は環境汚染の心配が全くない、次代のための安心の基礎素材。世界各地で産出する黒鉛は産地によってその性質が異なり、用途に適した使い方が必要です。

黒鉛の産地
GRAPHITE PRODUCING AREAS

●ウクライナ UKRAINE

黒鉛の分類
CLASSIFICATION OF GRAPHITE

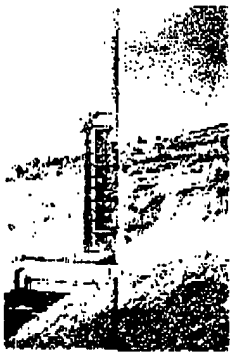
- 黒鉛 Graphite
  - 天然黒鉛 Natural Graphite
    - 鱗状黒鉛 Crystalline Graphite
      - 鱗片状黒鉛 Flake Graphite
        - マダガスカル MADAGASCAL
        - 中国 CHINA
        - ブラジル BRAZIL
        - ウクライナ UKRAINE
      - 鱗状黒鉛 Vein Graphite
        - スリランカ SRILANKA
    - 土状黒鉛 Amorphous Graphite
      - 韓国 R.KOREA
      - 北朝鮮 N.KOREA
      - 中国 CHINA
      - メキシコ MEXICO
  - 人造黒鉛 Synthetic Graphite

●マダガスカル MADAGASCAL

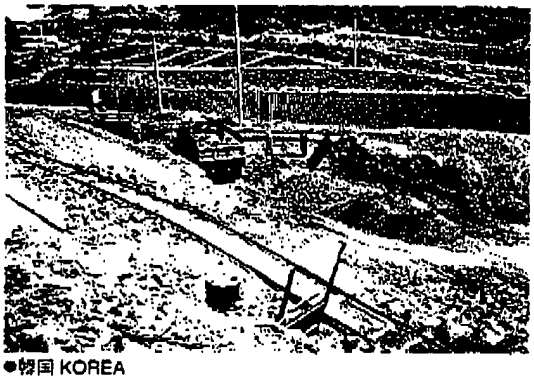
無限の可能性を秘めた
世界に求めて。

心の基礎
質が異なり、

A new world of graphite promising limitless possibilities for future generations. Graphite is a safe, next-generation basic raw material that poses no threat to the environment. Various kinds of graphite are produced throughout the world and each kind should be used for the most suitable application.



●中国 CHINA



●韓国 KOREA

●スリランカ SRILANKA

●ブラジル BRAZIL

個性いろいろ、可能性
黒鉛パワーのステージ

黒鉛は優れた特性をもった万能選手。あらゆる分野の技術革新を強力にサポートし、今後もその可能性の追求はますます拡がります。

■黒鉛の一般的特性

●分子量	12.011
●外観	黒灰色
●結晶系	六方晶系
●比重	2.23～2.26
●昇華点	3500℃
●硬度	1～2(モース)
●比熱	0.46(cal/g℃)
●熱伝導率	0.4～1.0(cal/cm sec℃)
●電気比抵抗	2～4×10⁻³Ω cm
●熱膨張係数	1.7×10⁻⁶l/℃
●弾性率	3～4×10³kg/cm²

■GENERAL PROPERTIES OF GRAPHITE

●Molecular weight	12.011
●Appearance	Blackish gray
●Crystal system	Hexagonal
●Specific gravity	2.23～2.26
●Melting point	3,500℃
●Hardness	1～2Mohs
●Specific heat	0.46cal/g℃
●Heat conductivity	0.4～1.0cal/cm sec℃
●Electric specific resistivity	2～4×10⁻³cm
●Coefficient of thermal expansion	1.7×10⁻⁶ l/℃
●Modulus	3～4×10³kg/cm²

黒鉛の性質
CHARACTERISTICS OF GRAPHITE

潤滑性
黒鉛は潤滑性に優れ、特に高温の雰囲気中、又は高荷重の場所においてもその特性はほとんど変化しません。メンテナンスフリーの潤滑法にも対応出来るため、その用途が拡がっています。

LUBRICATIVE PERFORMANCE
Graphite provides superb lubrication and its properties undergo almost no change even at high temperatures or under heavy loads. Thanks to its perfect suitability as a maintenance-free method of lubrication, the use of graphite is constantly expanding.

電気伝導性
黒鉛の電気比抵抗は2～4×10⁻³Ω cmで、銅、銀、金等の金属に比べやや劣るが、黒鉛は銅に比べ酸化されにくくかつ、銀、金に比べて安価なため広い範囲で使用されています。

ELECTRIC CONDUCTIVITY
Graphite's resistivity is 2～4×10⁻³Ω cm, which is a bit inferior to that of such metals as copper, silver and gold, but because graphite is less likely to be oxidized than copper and is cheaper than silver or gold, it is more widely used.

熱伝導性
熱の伝達メカニズムは電気と同様であるため、黒鉛は優れた熱伝導性を示します。使用例としては耐火物、ゴム、樹脂等に添加して熱伝導性の向上を図っています。

HEAT CONDUCTIVITY
Graphite exhibits the same heat conducting mechanism as electricity and offers excellent heat conductivity. In fact, it is added often to refractory, rubber, resin, and the like for the purpose of improving heat conductivity.

耐熱性
黒鉛は酸化雰囲気中では、約500℃以上の温度で酸化消耗が起こるが、非酸化雰囲気においては3,500℃まで安定であり、優れた耐熱性をもっています。製鋼用のマグネシアカーボン煉瓦をはじめ耐火物に多く使用されています。

HEAT RESISTANCE
Graphite is subject to oxidation wear at temperatures exceeding 500℃ in an oxidizing atmosphere but in a non-oxidizing atmosphere it remains stable up to 3,500℃ and offers excellent thermal resistance. It is commonly used in refractory materials, including magnesia carbon bricks for the iron & steel industry.

耐薬品性
黒鉛は炭素の共有結合性の結晶をもっており、非常に安定しています。従って酸、アルカリ両方の薬品に対して安定であり、高い耐薬品性を示しています。

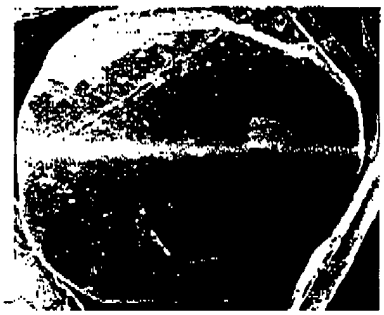
RESISTANCE TO CHEMICALS
Graphite possesses the covalent crystalline structure of carbon, which makes it extremely stable. It is stable in the presence of both acid and alkaline substances, and it has outstanding resistance to chemicals.

能性いろいろ。
ージは無限に拡がります。

新を強力に

Countless characteristics. Countless possibilities.
The saga of graphite is a story without end.
Graphite is a multi-purpose material featuring superior characteristics. It is destined to support tomorrow's technological advances and innovations in every field as its applications and its potential continue to grow.

黒鉛の種類
KINDS OF GRAPHITE



鱗片状黒鉛
外観が薄片状の結晶で最も黒鉛化の進んだ黒鉛です。

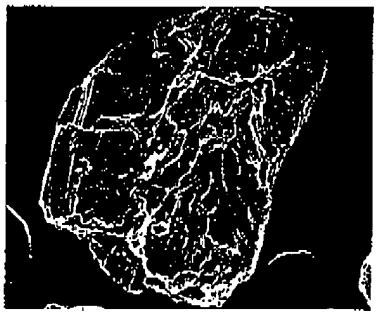
FLAKE GRAPHITE
This is the crystal having the foliated apperance and is the graphite of the most advanced graphitization.

鱗状黒鉛
脈状で産出する黒鉛で形状が塊状です。

VEIN GRAPHITE
This is the graphite produced in the vein state and having the massive shape.

土状黒鉛
外観が土状または土塊状を示す黒鉛で非晶質です。

AMORPHOUS GRAPHITE
This is the graphite having the earthy or massive appearance. It is amorphous.



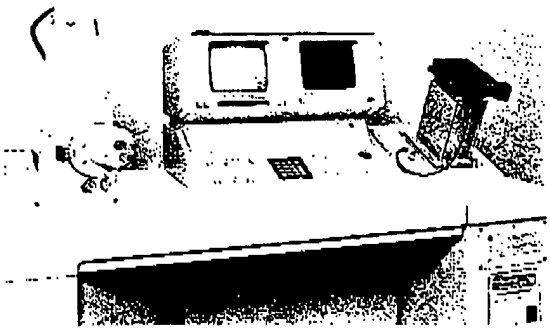
人造黒鉛
石油コークス等を原料として成型、焼成し更に2500℃以上の高温で黒鉛化されたものです。

SYNTHETIC GRAPHITE
This graphite is formed by molding raw materials such as petroleum coke, sintering the molding and graphitizing the sintered molding at a high temperature(above2500℃).

膨張黒鉛
天然鱗片状黒鉛を化学処理することにより、製造されたものが膨張黒鉛で、それを高温で加熱すると芋虫状(100～300倍)に膨張します。その膨張した黒鉛が膨張化黒鉛です。

EXPANDABLE GRAPHITE
A natural flake graphite which has been chemically treated and producing the expandable. It will expand (100 to 300 times) like a caterpillar when high temperature heated. It is the expanded graphite.

膨張化黒鉛
EXPANDED GRAPHITE



総合的エンジニアリ
あらゆるニーズにス

豊かな経験に支えられた信頼の技術と、先進システムから生まれる最新の技術で、あらゆるオーダーメイドに迅速にお応えします。

代表的な製品一覧表
A LIST OF OUR REPRESENTATIVE PRODUCTS

■CRYSTALLINE GRAPHITE

Type	F.C(%)	Ash(%)	Volatile matter(%)	Size		
CX-10000	99.0	0.5	0.5	Average	1μm	(3μm)
CX-3000	99.0	0.5	0.5	Average	2μm	(5μm)
FBF	99.0	0.5	0.5	Average	4μm	(7μm)
BF	99.0	0.5	0.5	Average	5μm	(8μm)
CBR	99.0	0.5	0.5	Average	15μm	(18μm)
SSC-3000	98.5	1.0	0.5	Average	2μm	(5μm)
SSC-600	98.5	1.0	0.5	Average	4μm	(7μm)
SSC-3	98.5	1.0	0.5	Average	5μm	(8μm)
SSC	98.5	1.0	0.5	Average	15μm	(18μm)
CX-600	97.0	2.0	1.0	Average	4μm	(7μm)
CPF-8	97.0	2.0	1.0	Average	5μm	(8μm)
CPF-3	97.0	2.0	1.0	Average	7μm	(10μm)
CPB-6S	97.0	2.0	1.0	Average	9μm	(12μm)
CPB	96.5	2.5	1.0	Average	15μm	(18μm)
96E	96.0	3.0	1.0	Average	7μm	(10μm)
96L	94.0	5.0	1.0	Average	7μm	(10μm)
96L-3	94.0	5.0	1.0	Average	15μm	(18μm)
90L-3	90.0	8.0	2.0	Average	15μm	(18μm)
CPC	87.0	11.0	2.0	Average	12μm	(15μm)
S-87	85.0	13.0	2.0	Average	15μm	(18μm)
K-3	83.0	15.0	2.0	Average	7μm	(10μm)
CF-80	99.0	0.5	0.5	+180μm	70%	
CF-48	99.0	0.5	0.5	+300μm	70%	
CF-32	99.0	0.5	0.5	+500μm	70%	
CP-150	98.0	1.0	1.0	−63μm	65%	
CP-100	98.0	1.0	1.0	45~150μm	75%	
HF-80	98.5	1.0	0.5	+180μm	70%	
HF-48	98.5	1.0	0.5	+300μm	70%	
HF-32	98.5	1.0	0.5	+500μm	70%	
SC-120	83.0	15.0	2.0	−150μm	90%	
SC-80	83.0	15.0	2.0	+150μm	60%	
SC-60	83.0	15.0	2.0	+250μm	60%	
SC-32	83.0	15.0	2.0	+500μm	70%	

■AMORPHOUS GRAPHITE

Type	F.C(%)	Ash(%)	Volatile matter(%)	Size		
APF-3000	92.5	5.5	2.0	Average	0.5μm	(3μm)
APF	92.5	5.5	2.0	Average	0.8μm	(6μm)
AX-600	83.0	14.5	2.5	Average	0.6μm	(4μm)
S-3	83.0	14.5	2.5	Average	0.7μm	(4μm)
AP-6	83.0	14.5	2.5	Average	2.0μm	(10μm)
AP-3	83.0	14.5	2.5	−45μm	95%	
300F	80.0	17.5	2.5	−45μm	90%	
150F	80.0	17.5	2.5	−104μm	90%	

アリングで、
こスピーディーに対応。

から生まれ
えします。

Responding to your needs with comprehensive engineering.
With reliable technology proven by a wealth of experience and leading-edge technology born of advanced systems, we can promptly meet your every need and requirement.

■SYNTHETIC GRAPHITE

Type	F.C(%)	Ash(%)	Volatile matter(%)	Size		
RA-10000	99.9	0.1	–	Average	1μm	(3μm)
RA-3000	99.9	0.1	–	Average	2μm	(5μm)
RA-15	99.9	0.1	–	Average	5μm	(8μm)
RA-44	99.9	0.1	–	Average	15μm	(18μm)
GX-600	99.5	0.5	–	Average	4μm	(7μm)
G-6S	99.5	0.5	–	Average	6μm	(8μm)
G-3	99.5	0.5	–	−75μm	90%	
G-150	99.5	0.5	–	−104μm	80%	
G-100	99.5	0.5	–	−150μm	80%	
G-48	99.5	0.5	–	0.3~0.15mm	80%	
G-30	99.5	0.5	–	0.5~0.104mm	80%	
G-50	99.5	0.5	–	2~0.3mm	80%	

■CALCINED COKES

Type	F.C(%)	Ash(%)	Volatile matter(%)	Size		
CMW-6S	99.5	0.5	–	Average	3μm	(6μm)
CMW-350	99.5	0.5	–	−45μm	90%	
CMW-200	99.5	0.5	–	−75μm	90%	
CMW-30	99.5	0.5	–	0.5~0.104mm	80%	

■SPECIAL TREATED GRAPHITE

Type	Ash(%)	Specific Surface	Size		
BSP-3000	2	58	Average	2μm	(5μm)
BSP-600	2	33	Average	5μm	(8μm)
BSP-3	2	27	Average	15μm	(20μm)

※( )内はレーザー回折法による測定値

μm	32	38	45	63	75	104	150	180	250	300	355	500	1000
Mesh	440	390	330	235	200	150	100	83	60	50	42	30	16

SPECIAL TREATED GRAPHITE

Flake graphite is widely used in various industrial products, including abrasive materials, batteries, and refractories, thanks to its excellent lubricity, electric conductivity, heat conductivity, heat resistance, and resistance to chemicals. However, flake graphite has poor compatibility with its counterpart materials, whether powder or liquid, and when mixed in, may cause various negative effects. For this reason, we developed special treated graphite with better compatibility than flake graphite.

PHOTO1 SPECIAL TREATED GRAPHITE (×200)

PHOTO2 FLAKE GRAPHITE (×200)

1 HOW SPECIAL TREATED GRAPHITE IS MADE

Special treated graphite is made from expanded graphite. First, flake graphite is treated with concentrated sulfuric acid and a strong oxidant such as hydrogen peroxide, hydrochloride peroxide, or manganate peroxide, so that graphite intercalation compounds are formed, which are then washed with water and dried to make expandable graphite. When the expandable graphite is heated at a high temperature of 950˚ to 1100˚C, the sulfuric acid or sulfuric ions inserted between the graphite layers are vaporized, the pressure generated by that vaporization spreads through the graphite, and the interlayer space of the graphite instantly expands 100 to 300 times. This product is then pulverized by a special method to make special treated graphite.

TABLE 1 CHARACTERISTICS OF SPECIAL TREATED GRAPHITE AND FLAKE GRAPHITE

	SPECIAL TREATED GRAPHITE	FLAKE GRAPHITE
Fixed carbon(%)	98.59	99.50
Ash(%)	0.39	0.11
Volatile matter(%)	1.02	0.39
Moisture(%)	0.40	0.20
Specific surface(m^2/g)	25.3	3.9
Bulk density(g/cm^3)	0.085	0.235
Particle size(μm)		
D_{10}	10.24	11.02
D_{50}	40.06	41.66
D_{90}	89.33	90.75

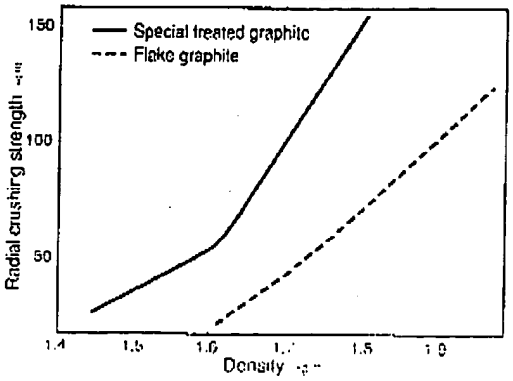
2 POWDER CHARACTERISTICS OF SPECIAL TREATED GRAPHITE

Special treated graphite has an extremely larger specific surface but smaller bulk density than scale graphite. SEM images of the two types of graphite are shown in Photo 1 and Photo 2. The special treated graphite has a honeycomb structure on the particle surface and thinner walls. In Figure 1, its radial crushing strength is compared to that of graphite molded by the no-binder method. Special treated graphite is extremely stronger than flake graphite.

3 USAGE RECORD OF SPECIAL TREATED GRAPHITE

It has proven highly effective when used in abrasive materials, batteries, refractories, motor brushes and other molded products. Applications in other fields are also increasing.

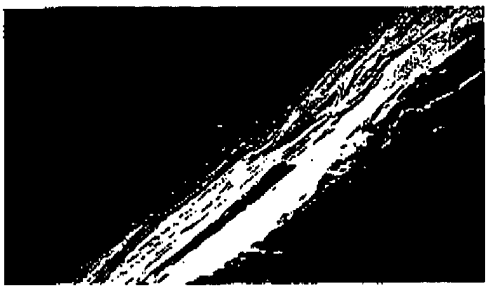
FIG.1 RADIAL CRUSHING STRENGTH OF GRAPHITE MOLDED



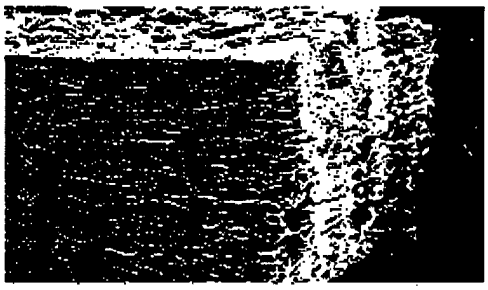
CHUETSU GRAPHITE WORKS CO.,LTD. TEL (05168[illegible] 3555 FAX (05168[illegible]6490 TEL (03 3255 5[illegible] FAX (03 3255 5[illegible]

EXPANDABLE GRAPHITE

Expandable graphite is natural flake graphite obtained by chemically treating graphite and heating it at a high temperature, so that it expands like (100 to 300 times). This unique characteristic makes it ideal for use in flame-resistant materials and insulating materials. By compressing and molding expanded graphite by the no-binder method, graphite sheet and graphite molded products can also be obtained.



EXPANDABLE GRAPHITE (×350)



EXPANDABLE GRAPHITE WITH EXPANDING (×350)

1 PRODUCTION OF EXPANDABLE GRAPHITE

Expandable graphite is produced by treating natural flake graphite with a concentrated sulfuric acid and nitric acid as well as with strong oxidizing agents (perchloric acids, perchlorates, dichromates, and hydrogen peroxides) , then rinsing and drying the compounds.

2 A LIST OF PRODUCTS

Type	Ash(%)	Specific Volume (cm³/g)	Size(Mesh)			
			+20	20~42	42~80	−80
			%	%	%	%
SSLF	0.5	200	5	70	20	5
SSMF	0.5	180	--	40	50	10
SSFF	0.5	180	−	20	70	10
SLF	1.0	200	5	70	20	5
SMF	1.0	180	−	40	50	10
SFF	1.0	180	−	20	70	10
ELF	5.0	180	5	70	20	5
EMF	5.0	180	−	40	50	10
EFF	5.0	180		20	70	10
CMF	8.0	150	−	40	50	10

3 METHOD FOR MEASURING SPECIFIC VOLUME (OUR STANDARDS)

(1)Maitain the moisture of the sample at 1% or under in the atmosphere of 80 to 110℃ in an electrothermostat.
(2)Collect 1g of the sample.
(3)Sufficientry heat a quartz measuring cylinder (bore 74mm ø and capacity 300cc) in the atmosphere of 950 to 1000℃ in an electric furnace.
(4)Quickly remove the quartz measuring cylinder in the electric furnace and put it into the cylinder.
(5)Again put the quartz measuring cylinder into the electric furnace and wait for about 30 seconds for the sample to expand fully.
(6)Gently remove the quartz measuring cylinder and allow it to cool down to room temperature, then precisely measure the weight of the sample.
(7)Lightly level off the top surface of the expanded sample by using a needle-shaped bar and measure the volume.
(8)Specific volume is determined as follows:

v=V/W v:specific volume
V:volume of sample after expansion(cm³)
W:waight of sample after expansion(g)

CHUETSU GRAPHITE WORKS CO.,LTD. TEL (06)681 2555 FAX (06)681 6290 TEL (03)3255-5594 FAX (03)3255-5596

Make the Innovation
株式会社中越黒鉛工業所
本　社 〒559大阪市住之江区平林北1-2-27
TEL(06)681-2555 FAX(06)681-6490
東京営業所 〒101東京都千代田区神田紺屋町1(商村ビル)
TEL(03)3255-5691 FAX(03)3255-5696